# RPS DEGREE COLLEGE

**BALANA (MAHENDERGARH)-123029**


# Lab Manual

***Chemistry (B.Sc. Hons. 1st & 2nd Semester)***

# Department of Chemistry

**ORGANIC CHEMISTRY**

**B.Sc. Hons. Ist Year**

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To prepare and purify through crystallization or distillation and ascertaining their purity through melting point or boiling point.

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3. P-Bromoacetanilide from acetanilide
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**SEMESTER 2**

Preparation and purification through crystallization or distillation and ascertaining their purity through melting point or boiling point.

1. Phenyl benzoate from phenol and benzoyl chloride
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# SEMESTER 1

# EXPERIMENT - 1

**Aim :** To Prepare pure sample of Iodoform

**Theory:**

Iodoform ($CHI_3$) is the iodine analogue of chloroform. It is a pale yellow crystalline solid (m.p. 119°C), having a characteristic odour. It is used as a mild antiseptic and disinfectant. It is also used in the preparation of many medicinal ointments used as pain-relievers.

lodoform can be prepared by treating any organic compound containing $CH_3CH(OH)$ - group (e.g., ethanol, 2-propanol, 2-butanol) or $CH3CO^-$ group (e.g., propanone, 2-butanone) with iodine in presence of sodium hydroxide. In the laboratory, it is usually prepared from either ethanol or propanone. The chemical reactions involved are:

**(a) With ethanol**

$2NaOH + I_2 \rightarrow NaOI + NaI + H_2O$

$CH_3CH_2OH + NaOI \rightarrow CH_3CHO + NaI + H_2O$

$CH_3CHO + 3NaOI \rightarrow I_3CCHO + 3NaOH$

$I_3CCHO + NaOH \rightarrow CHI_3 + HCOONa$

**(b) With Acetone**

$CH_3COCH_3 + 3NaOI \rightarrow CH_3COCI_3 + 3NaOH$

$CH_3COCI_3 + NaOH \rightarrow CHI_3 + CH_3COONa$

**PROCEDURE :-**

(i) Dissolve 5 g of iodine in 5 ml acetone or ethanol in a 100 ml conical flask or round bottomed flask.

(ii) Add 5% NaOH solution in small portions with constant shaking the flask. Cool the flask from time to time under tap water so that temperature does not rise above 40°c. The addition of NaOH solution is further continued till the brown colour of iodine just disappears.

(iii) Allow the flask to stand at room temperature for 5-10 minutes.

(iv) Filter the iodoform, wash with little cold water and then dry on a filter paper.

(vi) Recrystallize the crude iodoform by addition of small amount of rectified spirit in a 100 ml conical flask and heat it on a water bath.

(vii) Add more rectified spirit slowly till the iodoform dissolves.

(viii) Filter the solution quickly through a fluted filter paper into a beaker.

(ix) Cool the solution in ice. The iodoform will crystallize rapidly.

(x) Filter the crystals on a Buchner funnel, dry the crystals.

**RESULT:-**

(i) Yield of crystals = .....g
(ii) Colour of crystals = Sparkling yellow
(iii) Melting point = 119°c

# EXPERIMETN:- 2

**AIM:-** To prepare a sample of m-Dinitrobenzene from nitrobenzene.

**APPARATUS REQUIRED:-**

Round bottom flask, Air condenser, Water bath, glass rod, boiling chips.

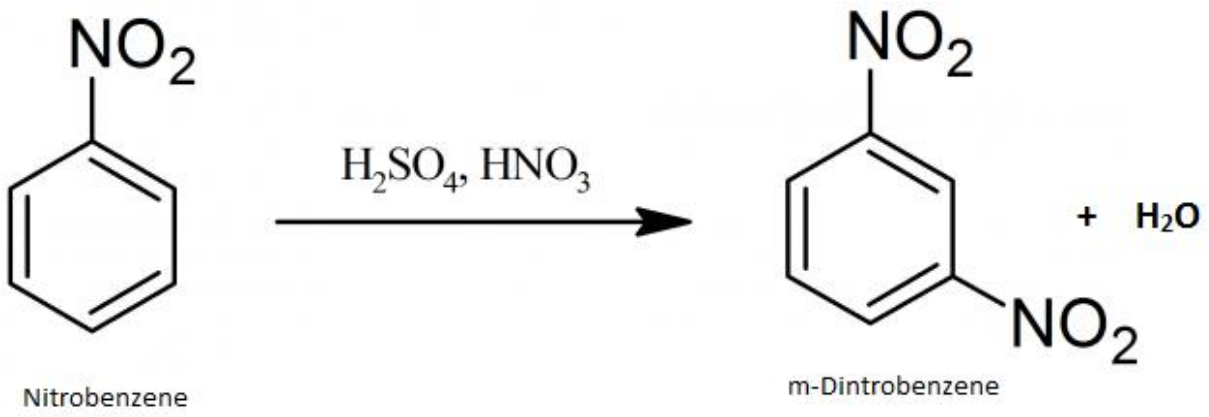
**CHEMICAL REQUIRED:-**

Nitrobenzene 5ml

Conc. $HNO_3$ 6-7ml

Conc. $H_2SO_4$ 10ml

**CHEMICAL REACTION:-**




**PROCEDURE:-**

i. Take 10ml of conc. $H_2SO_4$ and 6-7 ml of conc. $HNO_3$ in a 100ml round bottom flask.
ii. Add few boiling chips in the flask. Add nitrobenzene slowly with shaking and cooling the flask thoroughly.
iii. When whole of the nitrobenzene has been added shake the flaks vigorously fit it with air condenser and then place it on a boiling water bath.
iv. Clamp both the neck of the flask and condenser.
v. Remove and shake the flask time to time.

vi. After half an hour remove a drop of reaction mixture by means of glass rod and pour it into cold water. If it solidifies to a pale yellow solid, stop heating, remove the condenser and pour the reaction mixture from the flask into about 200 ml of cold water with continuous stirring when a yellow solid is formed.

vii. Filter the precipitates through a fluted filter paper. Wash it with cold water and dry it completely.

viii. Purification: add the precipitates in 25 ml of rectified spirit and heat the flask on a boiling water bath until the solid has completely dissolved. Filter while hot through a filter paper and dry them on a porous plate.

**PRECAUTIONS:-**

i. Add nitrobenzene in small installments and shake the flask thoroughly after each addition.

ii. Cool the flask in water if it becomes hot after each addition.

**RESULT:-**

i. Yield of crystals = …… g

ii. Colour of the crystals = …….

iii. Melting point = 90°C

# EXPERIMENT- 3

**AIM:-** To Prepare a sample of p-Bromoacetanilide from acetanilide.

**APPARATUS REQUIRED:-**

Conical flask, beaker, glass rod, burette etc.

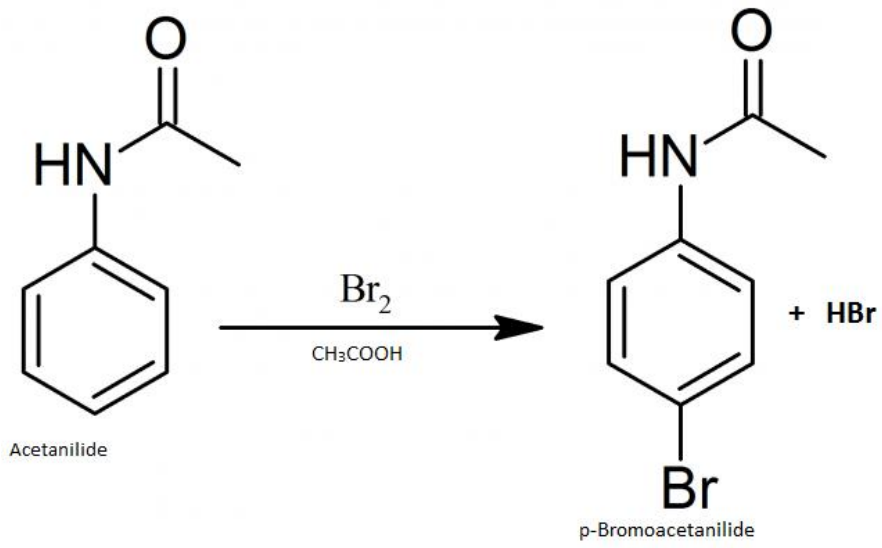
**CHEMICAL REQUIRED:-**

Acetanilide 4g

Glacial acetic acid 20ml

Bromine 1.8ml

**CHEMICAL REACTION:-**




**PROCEDURE:-**

i. Take 4 g of acetanilide into a 100 mL conical flask Add 10 mL of glacial acetic acid.Stirring with a glass rod may be necessary to dissolve the acetanilide.

ii. Now add 1.8 ml of bromine into 10 ml of acetic acid add bromine-acetic acid solution to acetanilide solution with stirring then leave the mixture 15 min.

iii. Transfer the mixture into beaker contain 100 ml of water with stirring. Collect the product by vacuum filtration using Büchner funnel.

iv. Purify the product by crystallization method using ethanol. Collect the white crystals by vacuum filtration, dried and weigh and calculate the percent yield.

**PRECAUTIONS:-**

i. Wear gloves and goggles during performing the experiment.
ii. Use extreme caution. Bromine burns can be quite severe.

**RESULT:-**

Yield- ……

Melting point - 167°C

# EXPERIMENT- 4

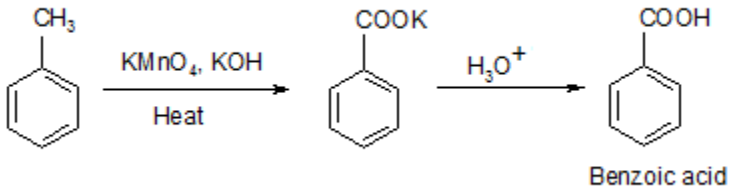
**AIM:-** To Prepare a sample of Benzoic acid from toulene.

**REQUIREMENTS:**

Potassium permanganate, KOH, toluene ,reflux condenser, filter paper ,ether etc.

**THEORY**

The reaction is simple oxidation where Methyl group gets converted to carboxylic group by the use of potassium permanganate as an oxidizing agent. Potassium hydroxide is used to provide alkaline medium because strong oxidizing agent Mn+7 is stable only in alkaline medium. Under neutral conditions Mn+7 is reduced to Mn+4 and under acidic conditions it is reduced to Mn+2; both of which are less powerful oxidizing agents. Added HCl first neutralizes the added KOH and after that converts potassium benzoate (present in soluble form in filtrate after reflux) into benzoic acid.




**PROCEDURE**

Mix Toluene (3 ml), Potassium Permanganate (10 grams) and dilute solution of sodium hydroxide in water (20 ml) in round bottom flask and set up the reflux. Allow mixture to reflux for 3 to 4 hours until oily toluene disappears. Then cool down the solution and filter the manganese oxide from the solution. Now addition of concentrated HCl to the filtrate will give you the precipitate of benzoic acid . You can either recrystallize it with hot H2O, or can extract the benzoic acid with ether.

**RESULT:-**

Yield- ……

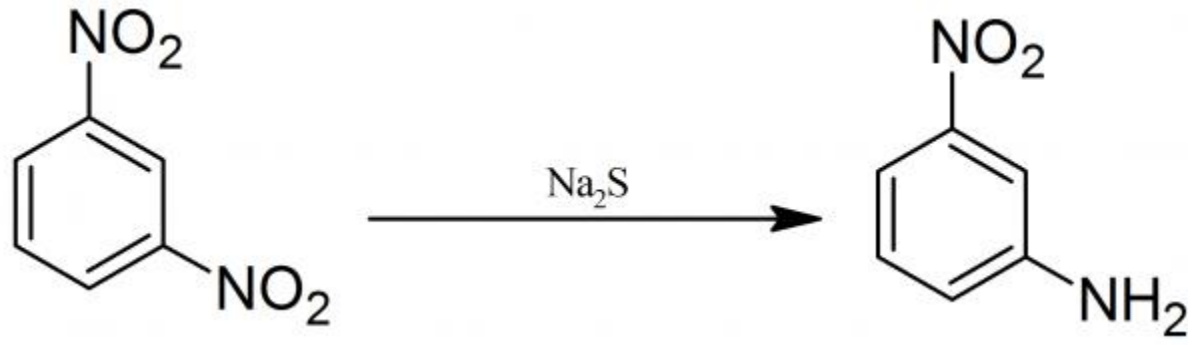
Melting point - 122.3°C

## EXPERIMENT- 5

**AIM:-** To Prepare a sample of m-nitroaniline from m-dinitrobenzene.

**REQUIREMENTS:** m-dinitrobenzene, ethanol ,ammonium hydroxide ,hydrogen sulphide ,beaker etc.

**THEORY:**




**PROCEDURE**

10 g of m-dinitrobenzene is dissolved in 40 g of ethanol and the solution is cooled down, upon which a portion of the m-dinitrobenzene separates out. This solution is further treated with 8 g of concentrated ammonium hydroxide solution (each 1 g of m-dinitrobenzene requires 0.8 g of concentrated ammonium hydroxide solution). The reaction flask with its content is tared, the mixture is saturated with hydrogen sulfide at room temperature and after saturation is complete the flask is heated under reflux for 30 minutes. Then the reaction flask content is cooled to room temperature and hydrogen sulfide again passed into it to solution until there is an increase of 6 g in weight (or 0.6 g for every gram of m-dinitrobenzene used). If correct weight is not reached hydrogen sulfide is again passed into the mixture. The solution is diluted with water, the precipitated crude 3-nitroaniline is filtered, washed with water, and extracted by warming with dilute hydrochloric acid. 3-Nitroaniline hydrochloride is converted to 3-nitroaniline base by neutralizing with ammonium hydroxide. Finally 3-nitroaniline is recrystallized from water yielding 70-80% of product which melts at 114° C.

**RESULT:-**

Yield- ……

Melting point - 114°C

## EXPERIMENT:- 6

**AIM:-** To purify a given sample of phthalic acid by sublimation.

**APPARATUS REQUIRED:-**

China dish, funnel, tripod stand, wire gauze, cotton.

**CHEMICAL REQUIRED:-**

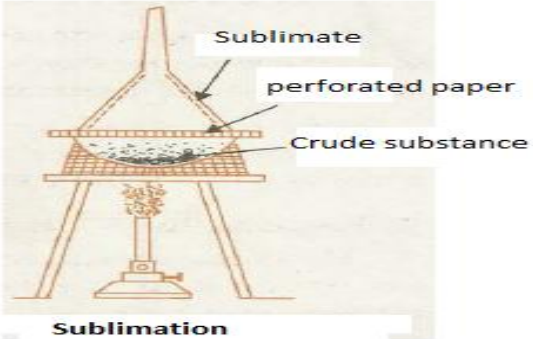
Impure phthalic acid - 5g

**THEORY:-**

Substances, which vapourise on heating, are purified by the method of sublimation. The substance, which has to be purified, is taken in a china dish, covered by a funnel. The china dish is heated on wire gauze. The substance volatilizes and the vapour condenses on the cooler portions of the funnel.

**PROCEDURE:-**

i. Take about 5g of impure phthalic acid in a dry and clean china dish and place it on a wire gauze kept on the tripod stand.
ii. Cover the china dish with a perforated paper and place and inverted funnel on it. Close the stem of the funnel with cotton.
iii. Heat the china dish on a low flame. Phthalic acid sublimes and condense on the cooler portions of the funnel.
iv. Remove the burner when whole of phthalic acid sublimes.
v. Cool and remove the funnel. Scratch pure phthalic acid from the inner walls of the funnel with a spatula on a watch glass.



Sublimation

# EXPERIMENT:- 7

**AIM:-** To purify a given sample of camphor by sublimation.

**APPARATUS REQUIRED:-**

China dish, funnel, tripod stand, wire gauze, cotton.

**CHEMICAL REQUIRED:-**

Impure camphor - 5g

**THEORY:-**

Substances, which vapourises on heating, are purified by the method of sublimation. The substance, which has to be purified, is taken in a china dish, covered by a funnel. The china dish is heated on wire gauze. The substance volatilizes and the vapour condenses on the cooler portions of the funnel.

**PROCEDURE:-**

i. Take about 5g of impure camphor in a dry and clean china dish and place it on a wire gauze kept on the tripod stand.
ii. Cover the china dish with a perforated paper and place and inverted funnel on it. Close the stem of the funnel with cotton.
iii. Heat the china dish on a low flame. Impure camphor sublimes and condense on the cooler portions the funnel.
iv. Remove the burner when whole of camphor sublimes.
v. Cool and remove the funnel. Scratch pure camphor from the inner walls of the funnel with a spatula on a watch glass.

# SEMESTER 2

## EXPERIMENT - 1

**Aim : To prepare Phenyl benzoate from phenol and benzoyl chloride.**

**Apparatus/Glassware Required:** 250 mL conical flask, beaker, volumetric flask, measuring cylinder, suction pump, Buchner funnel, filter papers etc.

**Chemicals Required**: Phenol, Benzoyl chloride, sodium hydroxide, alcohol

**Principle:** Phenols react with an aromatic acid chloride in the presence of excess of NaOH at room temperature to form an ester. The reaction is called Schotten Baumann reaction. If phenol is shaken with benzoyl chloride and excess amount of sodium hydroxide solution, it is benzoylated to give the ester, phenyl benzoate. The phenol is first converted into the ionic compound sodium phenoxide (sodium phenate) by dissolving it in sodium hydroxide solution. The phenoxide ion reacts more rapidly with benzoyl chloride than the original phenol does, but even so you have to shake it with benzoyl chloride for about 15 minutes. Solid phenyl benzoate is formed.

**Procedure:**

1. In a 100 mL conical flask dissolve 2.5 g of phenol in 35 mL of 10% sodium hydroxide solution and then add 5 mL benzoyl chloride to it.
2. Cork the flask properly and shake the mixture vigorously until the smell of benzoyl chloride has disappeared.

3. The phenyl benzoate which separates is filtered off, washed with cold water, dried, and recrystallized from alcohol.

**Uses:**

1. It can be used in a variety of polyesters, which have applications in products from clothing to heavy industry.
2. It is considered an excellent starting material for the production of optical components, particularly high quality lenses for still and motion picture cameras.

**Note:** Colourless crystals; insoluble in water; M.P. 68° C

**Precautions:**

1. Benzoyl chloride is lachrymatory and should be handled with care under a FUME HOOD.
2. Phenol is not only toxic but will cause severe burns.

**RESULT:-**

Yield- ……

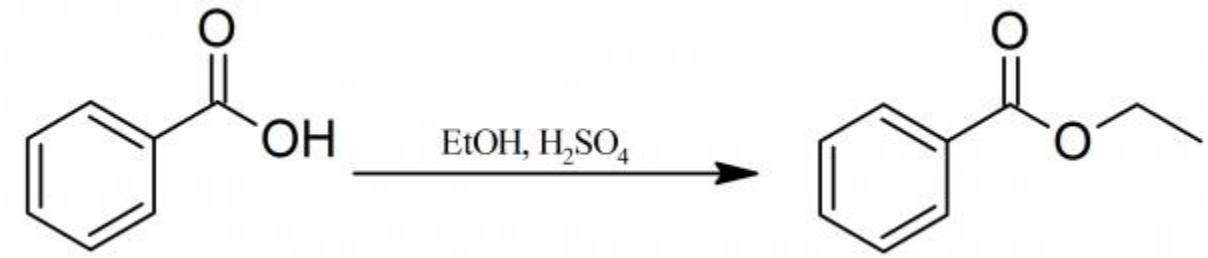
Melting point - 68°C

# EXPERIMENT - 2

**Aim: To prepare Ethyl benzoate from benzoic acid and ethanol.**

**REQUIREMENTS:** benzoic acid, ethanol, sulphuric acid, sodium carbonate, condenser, waterbath, ether etc.

**THEORY:**




**PROCEDURE:**

50 g benzoic acid are dissolved in 100 g absolute alcohol, 10 g concentrated sulphuric acid are added, and the mixture is boiled under a reflux condenser for 4 hours. Finally, about half the alcohol is distilled off on the waterbath, and the residue is diluted with 300 ml water, and neutralized with solid, powdered sodium carbonate, in order to remove all sulfuric acid and unchanged benzoic acid. The oil which has been separated is taken up with ether, the ethereal solution evaporated, and the residue dried over pure potassium carbonate and fractionated. The potassium carbonate should be prepared by heating pure potassium bicarbonate, and must be heated before use to render it quite anhydrous.

RESULT:

Boiling point of the ethyl benzoate, 212° C. Yield, 55 g.

# EXPERIMENT:- 3

**AIM:-** To prepare a sample of 2,4-Dinitrophenyl derivative of Acetophenone.

**APPARATUS REQUIRED:-**

Conical flask, beaker, glass rod etc.

**CHEMICAL REQUIRED:-**

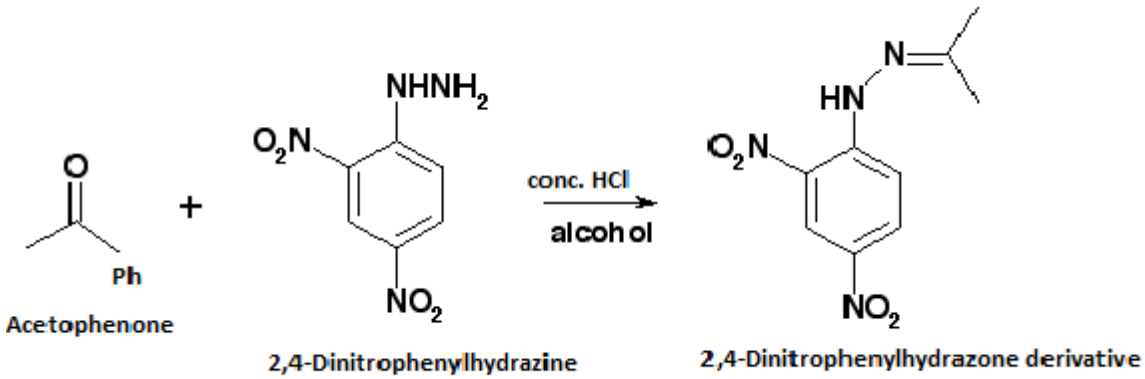
2, 4-DNP hydrazine - 1g

Acetophenon - 0.5g

Ethanol - 20ml

Conc. HCl - 2 ml

**CHEMICAL REACTION:-**




**PROCEDURE:-**

i. Add 1g 2, for –DNP in 20ml ethanol in a conical flask.
ii. Add 2ml conc. HCl and warm gently.
iii. Filter and add 0.5g acetophenone in the solution.
iv. Boil the solution and then cool it to room temperature.
v. Filter the crystals of 2, 4 DNP derivative and recrystallise them from ethanol.

**RESULT:-**

Colour of crystals - Orange

Yield - …..

M.Pt. - 237 -239°C